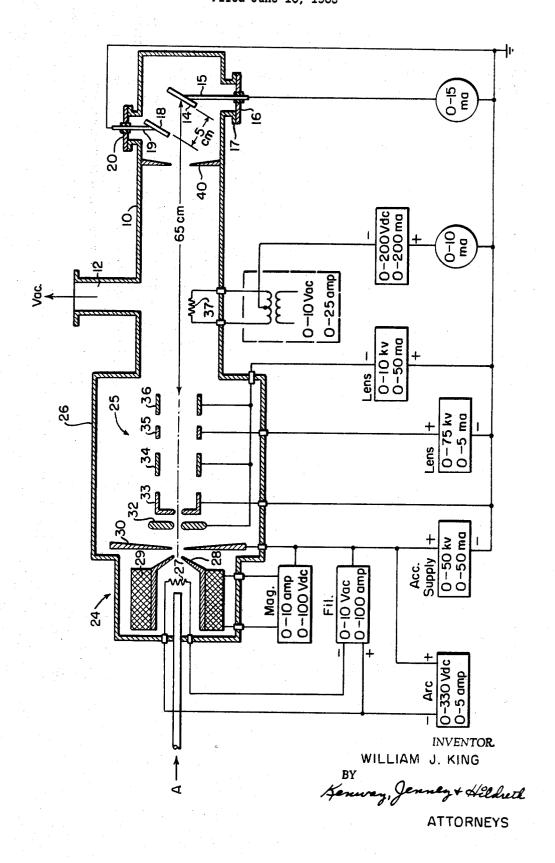
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METHOD AND APPARATUS FOR FORMING DEPOSITS ON A SUBSTRATE BY CATHODE SPUTTERING USING A FOCUSSED ION BEAM Filed June 16, 1965



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METHOD AND APPARATUS FOR FORMING DEPOSITS ON A SUBSTRATE BY CATHODE SPUTTERING USING A FOCUSSED ION BEAM
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9 Claims 10

#### ABSTRACT OF THE DISCLOSURE

A method and apparatus for forming a highly tenacious deposit of material on a substrate by directing a high 15 energy ion beam against the target, composed of the material that is to be deposited, in the presence of a vacuum of less than 10<sup>-5</sup> Torr to cause high energy particles of target material to be removed by sputtering and directing the removed, sputtered, particles against the substrate.

This invention relates to the formation of tenacious solid deposits in such forms as films, coatings, spots of the like.

Forming deposits of one material on the surface of another has many purposes, such as the formation of thin films for protective purposes; in the manufacture of capacitors, coated lenses; in the formation of passivating layers for semi-conductor devices; for establishing contact to make electrical connection or simply to join one material to another. Numerous of the available techniques are unsuitable for many purposes, such as chemical methods making useful solvents or electrolytes that would affect the surface to be coated, or methods requiring tem- 35 peratures which one or the other of the materials can not tolerate. A further trouble in many cases arises when the substrate to receive the deposit and/or the material to be deposited are of such nature that a strong bond is not easily established.

The present invention provides for the formation of a deposit of material which is highly tenacious, generally quite independently of the nature of the two materials, such that strongly adherent contact may be made between them and thin continuous films of one solid may 45 be applied to another.

In the process of this invention the material is transferred from a target source to the recipient substrate surface at high energies (5-50 kev. or more) and at low temperatures. Strongly adherent deposits may be built-up 50 under conditions which do not adversely affect either material.

The transfer of material is effected by directing a high energy ion beam against the target material, under a high vacuum of less than 10<sup>-5</sup> torr pressure, to cause particles 55 of the target material to be dislodged at high energy levels. By inclining the target surface to the ion beam the particles may be diverted to the side and deposited on a substrate surface located close by.

The process differs from known sputtering techniques 60 which require an appreciable pressure to provide an ionizable atmosphere in which emission of cathode material will take place. Material which is sputtered conventionally encounters many collisions with gas molecules in its path and does not strike the recipient surface at maxi- 65 mum energy, nor at right angles except by chance.

In the process of this invention all of the material removed from the target by the ion beam travels directly to the substrate surface. Because of the high vacuum few, if any, collisions with other particles are encountered, and 70 the particles accordingly hit the substrate surface at high energy and establish an extremely tenacious contact. It is

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assumed that the target material will in many cases penetrate or become embedded in the surface layer of molecules and become physically held to the recipient surface.

The process is applicable to a wide range of materials. Those which may be deposited include metals, metal oxides, non-metals and their oxides, and other compounds used for ceramics, dielectrics, semi-conductors, glass and

The substrate may be of almost any solid material, organic or inorganic, which is stable under high vacuum conditions. The process may be carried out cold, at room temperature or lower, or at elevated temperatures, as long as the vapor pressure is not too high for the required high vacuum condition that must be maintained.

Any of a number of accelerators capable of producing ion beams are known to the art as is their use to remove minute quantities of material, generally for analytical purposes. This invention is based on the discovery that these removal techniques, when carried out under very high vacuum, may be used to provide a tightly bonded thin film or other deposit on the substrate surface.

The apparatus of this invention consists of essentially an ion beam source mounted adjacent to a vacuum chamber, a target of material to be deposited mounted in the chamber in the path of the beam, with its exposed surface inclined to the beam, and means of supporting a closely spaced substrate material.

For the purposes of this invention the ion beam will consist of an inert gas, preferably one of the heavier gases, argon, krypton, or xenon, accelerated to an energy level suitable to cause material to be removed from the target surface.

The ion beam source should supply a beam of at least one to ten milliamperes at an energy level of preferably five to fifty kev., although some materials can be removed at lower energy levels. At higher energy levels, the sputtering coefficient drops off as the ions then tend to penetrate too deeply and become implanted into the target. Currents lower than one milliampere are suitable but the rate of film formation is correspondingly slow, and higher currents are more practical as long as overheating is avoided. The figures herein given are based on actual experience, and should not be taken as suggestive that the process does not operate outside the range given.

The preferred ion source is a duoplasmatron having a supply of argon for the ion beam, focused by an einzel electrostatic lens assembly. These are mounted to a vacuum chamber arrangement which carries the target and a holder for the substrate.

The apparatus and method of using it, which constitute this invention, will be better understood from the following detailed description and the accompanying drawing.

The preferred apparatus shown schematically in the drawing consists of a generally cylindrical chamber 10 which is maintained under a vacuum pressure of less than 10<sup>-5</sup> torr through a vacuum connection 12. The target 14 is mounted substantially axially within the chamber on a water cooled pedestal 15 carried by a plate 16 bolted to a flanged port 17. In a similar manner the substrate 18 is carried on a support 19 suspended from a plate 20 bolted to another flanged port 21.

The conducting supports 19 and 15 may be connected to the ground terminal as shown to carry away any resulting charge that would tend to accumulate on the target 14 or substrate 18 respectively.

The ion beam source is a duoplasmation 24 and an einzel lens system 25 mounted within a vacuum tight chamber 26 which connects with and forms part of the houing 10. In the duoplasmation, argon gas is introduced through a capillary to near the cathode 27 which is maintained at a moderate negative voltage, e.g., 100 volts, with respect to the anode, and is additionally heated by a

low voltage filament supply. Surrounding the cathode 27 is a solenoid 29 and intermediate electrode 28 which together serve to provide a magnetic field to confine the argon ion plasma in well known manner. The anode 30 having a central opening 31 is situated beyond the intermediate electrode, and in line with it and the cathode. The voltage between the anode and cathode causes the argon to ionize, forming a current conducting plasma of A+ ions. The energy level of the argon ion is controlled by the anode 30 which is typically maintained at 30 kv. 10 above ground. The argon ions are withdrawn from the duoplasmatron by the extraction electrode 32 and the negatively charged focusing electrodes 34 and 36 of the einzel lens, each of which is maintained at a moderate negative voltage, e.g., -1 to -2 kv., and are then focused 15 as a beam into the chamber 10, against the target 14. Focusing is accomplished by passing the ions withdrawn by the extraction electrode 32 through a ground electrode 33, the aforementioned negative electrode 34, which is an acceleration lens, a positively charged deceleration 20 lens electrode 35 maintained at a very high positive voltage, e.g., 15-25 kv., to condense the beam into a narrow ray, and the aforementioned second negatively biased acceleration lens electrode 36. The ion beam is controlled by adjusting the several voltages in well known manner. 25

Within the chamber and close by the path of the beam is mounted a heated fllament, e.g., of tantalum, which may be operated to neutralize the beam and prevent the accumulation of a charge on targets of dielectric material.

Additional control over the beam is provided by mount- 30 ing a target aperture 40, preferably made of material the same as the target, just in front of the target. This serves to protect the substrate material from ion bombardment, and further narrows the beam striking the target.

In a typical operation the housing is evacuated to a 35 pressure less than  $3\times10^{-6}$  torr, and the argon ion beam is accelerated to an energy level of 30 kev., and focused through the aperture 40, having a diameter of 1.6 centimeters. A narrow beam of several milliamperes is thereby caused to strike the surface of the target, which is 40 inclined to the beam at an angle a of 30 to 60°, preferably 45°. The substrate to be coated is mounted parallel to the target about 5 centimeters away.

The rates at which various materials have been found to deposit are given in the following table:

### POSITIVE ARGON IONS AT 30 KEV., $a=45^{\circ}$ , L=5 cm., $\phi=1.6$ cm.

A./minute/milliampere
Carbon 46
Aluminum 140
Silicon carbide 32
Titanium 57
Silicon dioxide 44
Titanium dioxide 14
Tantalum 45
Tungsten 31
where $\alpha$ equals the angle of inclination of the target to the beam: L equals the distance between the substrate and the target; $\phi$ equals the diameter of the aperture 40; and A. equals Angstroms.

Various films have been applied to several substrate 60 materials including glass, Mylar and various metals. It was noted that when either silicon or carbon was applied to a Mylar base, they adhered with such great tenacity and strength that the films could be wrinkled severely with negligible effect on the physical or electrical character- 65 istics of the film.

The application of a silicon dioxide film to a metal base followed by the application of a metal film provided a capacitor having a dielectric strength of about 5×10-6 volts per centimeter and a dielectric constant as high as 70 an energy sufficient to remove particles of said solid ma-

A characteristic of the process is the ability to deposit materials of extremely high purity. For instance, fused quartz may be applied by this process to the surface of a semiconductor to form a passivating layer.

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Although this invention has been described in detail with reference to the presently preferred embodiments, it is contemplated that modifications will occur to those skilled in the art and familiar with the principles herein disclosed, and that such modifications may be made without departing from the scope of the invention. In addition, while the invention is generally described as utilizing a beam of ions of an inert gas, it should be noted that the effect of the neutralizing filament 37 is to add electrons to the positively charged ion beam, to neutralize it, through the formation of atom particles. The formation of ions is necessary for electrostatic acceleration, but for bombardment purposes it is immaterial whether the particles are charged ions or neutral atoms.

Having thus disclosed my invention and described in detail the preferred embodiment thereof, I claim and desire to secure by Letters Patent:

- 1. The method of forming a tenacious deposit of a solid material on a solid substrate surface comprising directing a beam of ions of an inert gas along a selected path at a target surface of said solid material at an energy sufficient to sputter particles of said solid material at energies greater than thermal energies, placing said substrate surface in the path of said particles, substantially preventing the accumulation of any charge upon the target or substrate surface resulting from said beam and maintaining an ambient pressure less than 10-5 torr to prevent undue moderation of the energy of the sputtered
- 2. The method of forming a tenacious deposit of solid material on a solid substrate surface comprising ionizing an inert gas and forming a beam thereof, directing said beam thereof, directing said beam along a selected path, placing a target of said solid material in the path of said beam with a surface exposed to said beam inclined thereto, accelerating said beam to an energy sufficient to sputter particles of said solid material from said exposed surface at energies greater than thermal energies, placing said substrate surface spaced from said exposed surface in the path of said particles, substantially preventing the accumulation of any charge upon the target or substrate surface resulting from said beam and maintaining an ambient pressure below 10<sup>-5</sup> torr to prevent undue moderation of the energy of the sputtered particles and to permit a substantial portion of said particles to adhere to said substrate 45 surface and form a deposit thereon.
- 3. The method of forming a tenacious deposit of solid material on a solid substrate surface comprising ionizing an inert gas and forming a beam thereof, directing said beam along a selected path, placing a target of said solid 50 material in the path of said beam with a surface exposed to said beam inclined thereto, cooling said target to below its melting temperature accelerating said beam to an energy of between about 5 kev. and about 50 kev. to sputter particles from the surface of the target at energies greater 55 than thermal energies, placing said substrate surface spaced from said exposed surface in the path of said particles, substantially preventing the accumulation of charge upon the target or substrate surface resulting from the said beam and maintaining an ambient pressure below 10<sup>-5</sup> torr to permit a substantial portion of said particles to adhere to said substrate surface and form a deposit thereon.
- 4. The method of forming a tenacious deposit of solid material on a solid substrate surface comprising ionizing an inert gas and forming a beam thereof, directing said beam along a selected path, placing a target of said solid material in the path of said beam with a surface exposed to said beam inclined thereto accelerating said beam to terial from said exposed surface, neutralizing said beam to discharge the ions and form atoms thereof, placing said substrate surface spaced from said exposed surface in the path of said particles, and maintaining an ambient pres-75 sure sufficiently low to permit a substantial portion of

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said particles to adhere to said substrate surface and form a deposit thereon.

5. The method defined by claim 1 wherein the inert gas is selected from the group consisting of argon, xenon

6. The method defined by claim 1 wherein the solid material is selected from the group consisting of metals, metal oxides, non-metals, non-metallic oxides, ceramics,

dielectrics, semiconductors and glasses. 7. Apparatus for forming deposits of solid material 10 on a solid substrate surface comprising means for forming a beam of ions of an inert gas, means for directing said beam along a selected path, a support for material to be deposited in the path of said beam and inclined thereto, a support for the article to 15 receive said deposit adjacent to said beam and facing said inclined support, a housing providing a vacuum tight environment for said apparatus, means to form a vacuum of a pressure less than 10<sup>-5</sup> torr in said housing, a means whereby any charge upon the target or substrate surface 20 resulting from said beam is substantially prevented from accumulating, and means for maintaining said material at temperature below its melting temperature.

8. Apparatus for forming deposits of solid material on a solid substrate surface comprising means for forming 25 a beam of positive ions of an inert gas, means for directing said beam along a selected path, electron injection means adjacent to the path of said beam for neutralizing the ions therein, a support for material to be deposited in the path of said beam and inclined thereto, a support for 30 ALFRED L. LEAVITT, Primary Examiner the article to receive said deposit adjacent to said beam and facing said inclined support, a housing providing a vacuum tight environment for said apparatus, and means to form a vacuum of a pressure less than  $10^{-5}$  torr in said

housing.

9. Apparatus for forming deposits of solid material on solid substrate surface comprising means for forming a beam of ions of an inert gas, means for directing said beam along a selected path, a support for material to be deposited in the path of said beam and inclined thereto, means for cooling said support, a support for the article to receive said deposit adjacent to said beam and facing said inclined support, an elongated housing providing a vacuum tight environment for said apparatus, a means whereby any charge upon the target or substrate surface resulting from said beam is substantially prevented from accumulating, and means to form a vacuum of a pressure less than 10-5 torr in said housing said forming means comprising a duoplasmation, an anode positioned between said duoplasmation and said target, said directing means comprising an einzel lens positioned between said duoplasmation and said target, said support being inclined to said beam at an angle of greater than 30° but less than

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**35** 117—93; 118—49.1; 204—298; 313—63